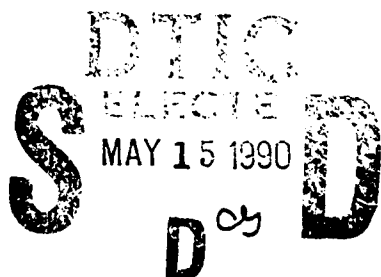

VIRGINIA TECH
CENTER FOR ADHESIVE AND SEALANT
SCIENCE

AN INTERDISCIPLINARY APPROACH TO THE PREDICTIVE
MODELING OF STRUCTURAL ADHESIVE BONDING

FINAL REPORT



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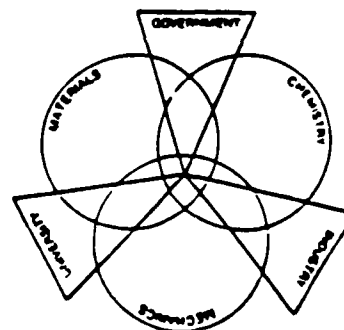
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FINAL REPORT

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**AN INTERDISCIPLINARY APPROACH TO THE PREDICTIVE
MODELING OF STRUCTURAL ADHESIVE BONDING**

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The Mechanics of Adhesive Joints

H. F. Brinson

Significant Results

The non-linear viscoelastic response of several commercial adhesives (FM 73, FM 300, et al) and other polymers (polysulfone, polycarbonate, et al) were extensively investigated in both bulk and bonded states. It was shown that several non-linear models (Schapery, Knauss, etc.) could be used to represent material response. Probably the most significant finding was the fact that the degree of non-linearity is not only a function of the stress scale but the time scale as well. This means that all short term testing and evaluation procedures may lead to non-conservative estimates of material response.

A new approach to the diffusion of moisture into polymers was developed and it was shown that mechanical properties have a coupled dependence on both stress and moisture. This approach and collaborating data provide new understanding of polymer behavior and offer the possibility of being able to predict delayed failures or the durability of adhesive bonds subjected to both stress and moisture with a precision not available previously.

A large amount of effort was expended in the development of finite element programs which could properly predict the response of bonded joints. The last code developed, NOVA, includes a non-linear constitutive model and our non-linear diffusion model as well. Thus, it is possible to predict the time dependent stress distribution of bonded joints under circumstances where the effects of stress and moisture intrusion are coupled.

A new double cantilever beam (DCB) test specimen geometry was developed which allows the measurement of the pure shear properties of an adhesive. This specimen has the advantage that it is easier to make than either the iosepescu or acan specimen. A closed form strength of materials solution was obtained and verified by the NOVA finite element code. Also, tests were performed to validate the stress analysis.

It was definitively established that mechanical properties of the interphase must be known in order for finite element models to be used to predict the durability or life of an adhesive joint. Efforts are proceeding to develop procedures for the evaluation of the mechanical properties of the interface layer. These include performing tests on a button specimen in an SEM and measuring deformations using a stereo imaging technique. It is anticipated that measurement of mechanical properties will be possible at the micron scale. Additional future work is anticipated to measure properties at the sub-micron domain. These efforts are continuing and will eventually focus on the DCB test specimen geometry.

Probably the most important aspect of all of the above is that each accomplishment was performed in an integrated interdisciplinary collaboration with colleagues in both materials science and chemistry. There is no question that our efforts have been enhanced by contributions from other fields and vice versa.

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Surface and Interphase Modification and Analysis

D. W. Dwight

Goals

With the eventual goal of using block copolymers as coupling agents, this year's work was aimed at understanding microphase separation in the bulk and surface segregation as a function of the following variables: block size, topology and chemical composition, solvent and evaporation rate, casting substrate and concentration in blends with homopolymer.

Significant Results

Polymethyl methacrylate with graft sidechains of polydimethyl siloxane showed a most interesting effect of block size and concentration. At ~50% siloxane of $mw = 10,000$ and $20,000$ a higher regular, rod-like morphology appeared in the bulk of thick cast films (using TEM on or with thin films, the morphology was spherodidal).

For the first time, we demonstrated that segregation of a low surface energy block is suppressed at substrate relative to the air surface. This was shown to be the case with sulfone/siloxane block copolymers cast on water, mercury and crosslinked polydimethylsiloxane. Also it was shown that evaporation rate has a marked effect upon surface segregation: the siloxane surface concentration was still increasing slowly after three day evaporation at room temperature. At the fastest evaporation (about 1 minute for thin films cast on water), siloxane of $mw = 9,000$ showed preferential segregation to the air surface while at $mw = 4,000$, air and water surfaces were identical.

In the poly(styrene/diene) triblock systems, SIS and SBS, preferential surface segregation of the diene block was determined, and moreover the $C = C$ linkages in the drenes were oriented away from the surface. However, those effects were eliminated by using the solvent toluene as opposed to THF.

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The Stress Analysis of Adhesive Joints by the Finite Element Method

J. N. Reddy

Primary Goal

The primary goal of this research effort is to gain an understanding of the process of adhesion, and predict the strength and reliability of adhesively bonded joints using the finite element method.

Significant Results

The most significant contribution of the research is the development of a finite-element computational procedure that accounts for geometric nonlinearity, Schapery's nonlinear viscoelastic constitutive model, and nonlinear moisture diffusion model. The procedure is validated for both geometrically nonlinear options for adherends by comparing the present results with those available in the literature. A systematic investigation is carried to determine the geometric nonlinearity, viscoelasticity, and moisture diffusion effects on the stress distributions in bonded joints.

In the two-dimensional finite-element analysis program, called NOVA, during this research the large displacements and rotations experienced by the adherends and the adhesive are taken into account by invoking the updated Lagrangian description of motion. The adhesive layer is modeled using Schapery's nonlinear single integral constitutive law for uniaxial and multiaxial states of stress. Effect of temperature and stress level on the viscoelastic response is taken into account by a nonlinear shift factor definition. Penetrant sorption is accounted for by a nonlinear Fickian diffusion model in which the diffusion coefficient is dependent on the penetrant concentration and the dilatational strain. The validity of the diffusion model is demonstrated by several numerical examples. A delayed failure criterion based on the Reiner-Weissenberg failure theory has also been implemented in the finite element code. A Users' Manual on the computer program 'NOVA' is also prepared and made available to interested people. The program could be coupled with a commercially available pre- and post-processor programs to analyze viscoelastic bonded joints, and predict stress and strain distributions and onset of failures.

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Stress Durability Testing of Thermoplastic/ Titanium Adhesive Bonds in Severe Environments

Thomas C. Ward

Significant Results

The durability of Ti/PES adhesive bonds is studied by stress durability testing (ASTM 2919), utilizing a "3M" durability tester. The testing apparatus consists of an immersion insulating/recirculating bath with capacity to test twenty-four (24) specimens simultaneously. The test is performed on single lap shear specimens (ASTM 1002). These are fastened to a vertical fixture and loaded in tension through an external spring. The load is applied with an hydraulic tensioning device, and then released. The fixtures are immersed in water at 80°C. The time to failure is recorded by switched hour timers. The single lap shear specimens are prepared positioning the Ti coupons and 1 sp.in. PES film in an aluminum alloy jig and are compression molded. Additional specimens are prepared incorporating 1 sp.in. Ti foil sheets between PES films to make a "sandwich" composite bond structure. The metal surface of the coupons and the foils was modified by chromic acid anodization (CAA). The creep results show a great degree of scatter. Also, it appears that the durability of these bonds increases with larger number of interphases. The scatter of the data can be attributed to the brittleness of the adhesive at test temperature. In this instance, processing procedures are considered critical. The apparent increase in durability with larger number of interphases indicates that the parameter of bond thickness should be controlled more carefully. That is, the test specimens must have a uniform total bond thickness for every bond structure. This aspect will be approached by compressing PES films of different thicknesses, and utilize a spacer in the jig to achieve a constant uniform bond thickness. The fracture surfaces will be characterized by X-ray Photoelectron Spectroscopy (XPS), Scanning Electron Spectroscopy (SEM) and FTIR Microspectroscopy. The composite bond will be characterized by Dynamic Mechanical Analysis (DTA). These analyses will aid in the prediction of the mechanical response of these bonds.

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Adherend Surface Characterization and Adsorption

J. P. Wightman

Primary Goal

The scientific goal of the research is the prediction of durability in metal-polymer adhesion. The specific goals of the research project are the physical and chemical analysis of Ti 6-4 adherend surfaces, the determination of the performance of pretreated Ti 6-4 adherends bonded with a high temperature epoxy and exposed to a hot-moist environment and the role of metal alkoxides in enhancing the durability of epoxy/Ti 6-4 bonds.

Significant Results

Pretreatments given to Ti-6-4 adherends created chemically and physically reproducible oxide surfaces. The anodized surfaces were porous; whereas, the etched surfaces were not. TURCO created a rougher surface than the P/F (phosphate-fluoride) and CAA (chromic acid anodized) surfaces. The work of adhesion between epoxy and Ti-6-4 oxides is thermodynamically predicted to decrease in a water environment.

The strength and durability of Ti-6-4/FM-300 epoxy bonds was tested with the lap shear and the wedge tests. FM-300 penetrated the porous oxides. The lap shear test was not sensitive to the surface pretreatment when tested in ambient conditions. However, when the lap shear was placed under 40% load and placed in hot, humid conditions, a clear differentiation between surface pretreatments was observed. By both the stress durability and the wedge test, the CAA and SIA (sodium hydroxide anodized) surfaces were most durable followed by the TURCO surfaces. The P/F surfaces showed the poorest durability. The locus of failure differed between the lap shear test and the durability tests.

Metal alkoxides were used to enhance the bond durability. Sec-butyl aluminum alkoxide (E-8385) significantly enhanced the bond durability of the P/F surfaces but leveled the durability of the CAA surface to that of the P/F coated with E-8385. The titanium alkoxides did not enhance the durability of P/F bonds. The locus of failure occurred within the primer layer. The aluminum alkoxide surface contained a higher concentration of hydroxyl groups than the titanium alkoxides.

One explanation for the differences in durability between the pretreatments is surface area. The porous oxides were the most durable and also contained the highest surface area. The TURCO surfaces were the roughest, therefore, increasing the surface area over that of the P/F surfaces. Moisture intrusion hydrolyzes physical bonds such as dipole-dipole, acid-base, hydrogen bonds. The kinetics of moisture intrusion is slower for the higher surface area surfaces, thus better durability. The metal alkoxide coated P/F samples, however, contain approximately the same surface area. But, the aluminum alkoxide is significantly more durable. The concentration of surface hydroxyls is higher, yielding a higher density of hydrogen bonds, thus better durability.

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